

Utilization of Low NO_x Coal Combustion By-Products

**Quarterly Report
January - March 1995**

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May 1995

Work Performed Under Contract No.: DE-FC21-94MC31174

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Michigan Technological University
Houghton, Michigan

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Office of Fossil Energy
Morgantown Energy Technology Center
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Morgantown, West Virginia 26507-0880

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1400 Townsend Drive
Houghton, Michigan 49931

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**UTILIZATION OF LOW NO_x COAL COMBUSTION BY-PRODUCTS
DE-FC21-94MC31174**

PROJECT SUMMARY - SECOND QUARTER

January 1 through March 31, 1995

TASK 1.0 TEST PLAN

This task has been completed.

TASK 2.0 LABORATORY CHARACTERIZATION

TASK 2.1 Sample Collection

Samples have been obtained from America Electric Power Company, Baltimore Gas & Electric Company, and Nevada Power Company. These companies use the three most popular low NO_x burners.

Nevada Power Company (NP) uses a FRONT-DRY BOTTOM burner, made by Foster Wheeler Energy Corporation, with a capacity of 2,090 mlb/hr. NO_x control is by boiler design. Boiler design constitutes about 13% of NO_x control technology and is the third largest NO_x control method. Nevada Power burns sub-bituminous, low sulfur coal from Utah, producing a Class C fly ash. Nevada Power Company is an EPRI member.

Fifteen 55-gallon drums of fly ash from Nevada Power were received by the IMP in January, 1995. Representative samples from each drum were collected and LOI values for these samples were determined. This was done to evaluate how the LOI content varied. A head sample was obtained by mixing the representative samples from all fifteen drums.

Baltimore Gas & Electric Company (BGE) uses an OPPOSED-DRY BOTTOM burner made by Babcock & Wilcox. The capacity of the burner is 4,425 mlb/hr. NO_x control is by staged combustion technology, the fifth largest (5.4%) NO_x control method. They burn bituminous, low sulfur coal from West Virginia and generate a Class F fly ash. Baltimore Gas & Electric Company is an EPRI member.

Fourteen 55-gallon drums of fly ash were received from Baltimore Gas & Electric Company in January, 1995. Representative samples from each drum were collected. A head sample was obtained by mixing the representative samples from each of drums.

America Electric Power Company (AEP) supplied ash from the Appalachian Power Company Glen Lyn Plant, which uses a single wall fired-dry bottom boiler made by Babcock & Wilcox. The capacity of the burner is 1,523 mlb/hr and NO_x control is by staged combustion technology. The plant burns eastern bituminous-low sulfur coal, resulting in a Class F fly ash with a high LOI. America Electric Power Company is an EPRI member.

Twenty three 55-gallon drums of fly ash were received from America Electric Power Company in January, 1995. Representative samples from each drum were collected and a head sample was obtained by mixing the representative samples from each of drums.

We are currently in contact with Detroit Edison, New York State Electric & Gas Corporation, and Public Service of Colorado in an effort to obtain three more low NO_x ash samples.

TASK 2.2 Material Characterization

For each as-received ash sample, the overall or head LOI value was determined. A particle size distribution of as-received fly ash was determined by sieve analysis for the +400 mesh fraction and Microtrac analysis for the -400 mesh fraction. The amount of magnetic material in the as-received ash is determined using a Davis Magnetic Tube Test.

The LOI value of Nevada Power ash samples from each of the 15 drums varied from 2.09% to 4.90%, with an average of 4.00% LOI. The preliminary test results show that the Nevada ash contains about 1.8% magnetic material. BGE as-received ash had an LOI value of 6.37% and AEP had a 22.21% LOI head grade. The magnetic analyses for these samples are not yet complete. The size distributions, with LOI by size data, are given in Appendix A.

The density of the three as-received ash samples were measured: 2.25 g/cm³ for NP ash, 2.24 g/cm³ for BGE ash and 2.13 g/cm³ for AEP ash.

Other characterization tests are underway.

TASK 2.3 Laboratory Testing of Ash Processing Operations

A summary of the laboratory results to date are given in Appendix B. The first two tests were conducted on NP ash, since this was the first received and NP themselves were interested in some preliminary results. A reasonably successful separation was obtained, although further testing is necessary.

The focus then shifted to the AEP and BGE ash samples. The BGE ash has a very high natural pH, which will require modification of the flotation scheme. The AEP ash is a Class F ash that is very

similar to samples studied extensively in previous projects, except for a much higher carbon content. The original intentions were to accelerate the laboratory testing of the AEP ash and move on into the pilot plant stage. The initial flotation test with AEP ash was very successful, however the results were not repeatable. Therefore, various parameters were evaluated, including use of a dispersant, various reagents for and levels of pH adjustment, different collector and frother reagents and dosages, and floating only the -100 mesh fraction of the sample. The best results to date for the AEP ash, and that which will be used in processing in the pilot plant, are a sizing step at 100 mesh to remove the coarse fly ash and coarse carbon, and approximately 10 pounds of collector (a fuel oil / vegetable oil mix) per ton of fly ash.

A series of laboratory tests (Tests 11-17) were run to evaluate the effect of percent solids on the flotation process. Samples of ash from a previous project (FA5, Class F ash from Consumers Power Company) were used since flotation parameters were well established for that material. The percent solids was increased from 8% up to 40%, keeping the cell volume, reagent pounds per ton, and other factors constant. Results indicated that it was possible to obtain acceptable LOI values (<2%) in the clean ash, even at 40% solids.

TASK 3.0 PILOT PLANT TESTING

The circuit has been assembled in preparation for the first pilot plant run, scheduled for the first week in April. The first run was conducted on AEP ash, since this has the highest carbon content and is similar to ash samples run previously for other projects. The results will be discussed in detail in the next quarterly report, in summary, the run went very well, yielding a clean ash product containing 0.26% LOI and 57% weight of the feed material.

Initial testing indicated that the use of different methods of drying the clean ash can have a broad effect on the pozzolanic nature of the ash produced. Arrangements are being made to allow the use of a drum filter and calciner for dewatering and drying of the clean ash product. The calciner is gas fired with two principle zones, each of which is fitted with three individual North American burners, thus offering a significant variation in the temperature profile. The unit allows for a broad variation in the mechanical configuration as well, providing extensive flexibility in the processing of the ash.

TASK 4.0 PRODUCT TESTING

TASK 4.1 Concrete Testing

Important characteristics of the as-received ash samples related to use in concrete are given in Table 1. These include % LOI, which has an ASTM C 618 limit of 6 wt% LOI, and a size restriction, measured by the percent weight of sample retained on a 325 mesh sieve, which is a maximum of 34

wt%. All three samples studied to date meet the size restriction, although the AEP ash is very close to the maximum allowed. Only the NP ash is under the ASTM % LOI allowed for use in concrete.

Table 1. Characteristics of Ash for use in Concrete				
	NP	BGE	AEP	ASTM *
% LOI	4.00	6.37	22.21	6.0
wt% retained on 325 mesh	19.4	19.5	32.9	34
* ASTM C 618 specification				

Test cylinders to determine the seven, twenty eight, and ninety one day compressive strength of 35S concrete samples with 8%, 20%, 30%, 40%, 50% and 60% cement replacement by as-received NP ash (Class C) have been made. Some seven day compressive strength data has been obtained.

Test cylinders to determine the seven, twenty eight, and ninety one day compressive strength of 35S concrete samples with 8%, 20%, 30% and 40% cement replacement by as-received BGE ash (Class F) and 8%, 20% and 30% cement replacement by as-received AEP ash have been made. Slump, air content, and the water to cementitious materials ratios were obtained for all tests.

TASK 4.2 Concrete Block/Brick

This subtask has not started.

TASK 4.3 Plastic Fillers

Subtask 4.3.1. Size Classification

Hydrocyclones and air classifiers are potential equipment for making a fly ash separation at a five micron cut size. Equipment manufacturers were contacted and an MTU professor with strong expertise in this area was also consulted. Four potential separation technologies were identified. First, based on telephone conversations with Carpc, Inc. and literature received, the Mozley 10 mm C1009 hydrocyclone seems to meet our requirements. The Mozley 10 mm hydrocyclone is a high performance, small diameter unit offering d_{50} cut points in the 2 to 5 micron size range and a maximum throughput of 0.25 m³/h per cyclone. In an industrial situation, many cyclones can be installed in one unit to treat high volume slurry. Second, the KC-20, manufactured by Cyclo-Tex, Inc., is an aerodynamic particle classification system, capable of a 5 micron cut and 500 lbs/hr production rate. Third, the Micro-Sizer

MS-5, manufactured by Progressive Industries, Inc., is an air classifying system designed to produce ultra-fine products down to a 3 micron top size with average particle diameters down to 0.4 microns. The feed rate for this system ranges from 100 to 2000 lbs/hr. Larger systems are also available, such as MS-100, capable of producing 50 to 100 tons/hr of ultra-fine powders. Fourth, the Turbo Classifier TC-60, manufactured by Nisshin Engineering Co., Ltd, is capable of a particle size cut at 2-50 microns and 1000 kg/hr output. Larger classifiers are also available. The information we have indicated that industrial classification systems are available to separate fly ash into fine (less than 5 microns) and coarse fractions at reasonable prices.

The laboratory classification facilities currently available at IMP include hydrocyclones and an air classifier. After a discussion among the research team, we will use the air classifier to produce the fine fly ash powder for plastic filler. This classifier (Model A-12 Acucut) was manufactured by Hosokama Micron Powder Systems. Currently, two small powder containers need to be replaced and have been ordered. After we receive the containers, the separation test will begin.

Subtask 4.3.2. Coating Test

The purpose of the coating test is to improve the brightness of clean fly ash. So far, TiO_2 , CaCO_3 , and ZnO coatings have been tested. These coatings were produced on fly ash surfaces by precipitating the products generated by the following possible reactions:

- 1) $\text{TiCl}_4 + 4\text{NaOH} = \text{TiO}_2 + 2\text{H}_2\text{O} + 4\text{NaCl}$
- 2) $\text{Ca}(\text{OH})_2 + \text{CO}_2 = \text{CaCO}_3 + \text{H}_2\text{O}$
- 3) $\text{CaCl}_2 + 2\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 = 2\text{NaCl} + \text{CaCO}_3 + 2\text{H}_2\text{O}$
- 4) $\text{ZnCl}_2 + 2\text{NaOH} = \text{Zn}(\text{OH})_2 + 2\text{NaCl}$

It has been determined that the TiO_2 and CaCO_3 coatings are transparent and can not improve fly ash brightness. The ZnO coating test is continuing.

TASK 4.4 Activated Carbon

This subtask has not started.

TASK 5.0 MARKET AND ECONOMIC ANALYSIS

During the last quarter, marketing efforts focussed on developing industrial partners to evaluate plastic extender materials under actual production conditions. The effort is more complex

than anticipated because of the nature of the plastic resin business. Major polymer producers, such as DOW and GE, provide material to formulators or to their own facilities that add fillers, lubricants, colorants, and a host of other additives to make unique blends for specific applications. Very quickly, a single polymer literally becomes hundreds of formulations. We are trying to get a formulation that first, is representative, and second, is complete except for the presence of filler.

End product producers consider their formulations proprietary and the polymer producer cannot provide an unfilled resin without knowing the formulation that a manufacturer is using; a catch 22 situation. We are overcoming these issues by developing close relationships with a few significant injection plastic product producers to obtain their formulation that in turn can be taken to the formulator or primary polymer producer to obtain samples of the same formulation complete except for extender material.

The solicitation of industrial participants has been conducted through a mailing to Michigan companies listed in the latest Michigan Plastics Industry Directory and from a compiled list of national plastic product trade associations. The solicitation letter requested assistance in four areas: 1. General review of the project research plan; 2. Sample quantities of molding formulations; 3. Guidance in selecting critical test protocols; and 4. Review of final results.

Several of the national associations have agreed to review the project research plan and to review final project results. Some of the organizations are circulating our request to their membership and encouraging them to participate in the project.

The responses to the Michigan solicitation include a manufacturer of filler enhanced injection molded polyethylene acoustical products, a manufacturer of glass filled polypropylene, the nation's largest cold molder of unsaturated polyethylene, and a producer/supplier of unique filler materials. All responses to the industrial participation solicitation have been followed up with a writeup of the project research plan.

Soo Plastics, a plastic parts injection molding firm from Sault Ste. Marie, Michigan, recently called to say that they would be forwarding samples of three materials, used frequently at their facilities, in which calcium carbonate has previously been used as a filler material. With calcium carbonate and ash displaying similar attributes, a successful substitution may be possible.

Effort is also continuing to establish the economics of the separation process. There is a great deal of testing yet to be carried out on each of the ash streams being generated before design parameters, for each of the key pieces of equipment used in the beneficiation process, can be established. Even so, a number of manufacturers of process equipment have been approached in an effort to establish a potential commercial operation. The companies include Denver Equipment Company, Dorr-Oliver, Filtra Systems, Eimco Inc., as well as others which have already offered assistance with regard to development of an economically viable operation.

APPENDIX A

TASK 2.2 Material Characterization

Size Distribution and LOI by size data

NEVADA POWER AS-RECEIVED ASH - SIZE DISTRIBUTION and LOI by Size							
Tyler Mesh		% Weight		% LOI		Distribution	
	Wt. (g)	individual	cumulative	individual	cumulative	individual	cumulative
+65	2.4	2.30	2.30	37.83	37.83	21.81	21.81
+100	1.2	1.15	3.45	24.53	33.40	7.07	28.88
+150	2.2	2.11	5.56	9.80	24.45	5.18	34.05
+200	4.2	4.02	9.58	4.72	16.16	4.76	38.82
+270	5.6	5.36	14.94	3.60	11.65	4.84	43.66
+325	4.6	4.41	19.35	2.92	9.66	3.23	46.88
+400	6.6	6.32	25.67	2.82	7.98	4.47	51.35
-400	77.6	74.33	100.0	2.61	3.99	48.65	100.0
head	104.4			3.99			

NEVADA POWER AS-RECEIVED ASH - MICROTRAC ANALYSIS		
Channel	Cumulative	Volume
62	100.0	2.6
44	97.4	9.1
31	88.3	11.4
22	76.9	12.0
16	64.9	11.7
11	53.3	10.2
7.8	43.1	9.6
5.5	33.5	7.5
3.9	26.0	9.2
2.8	16.7	9.6
1.9	7.2	4.0
1.4	3.2	2.5
0.9	0.7	0.7

BGE AS-RECEIVED ASH - SIZE DISTRIBUTION and LOI by Size							
Tyler Mesh		% Weight		% LOI		Distribution	
	Wt. (g)	individual	cumulative	individual	cumulative	individual	cumulative
+65							
+100	1.2	1.54	1.54	39.83	39.83	9.85	9.85
+150	2.2	2.82	4.36	19.06	26.39	8.64	18.48
+200	4.0	5.13	9.49	14.33	19.87	11.81	30.29
+270	4.6	5.90	15.38	10.23	16.18	9.69	39.98
+325	3.2	4.10	19.49	10.58	15.00	6.97	46.96
+400	8.4	10.77	30.26	6.76	12.07	11.70	58.65
-400	54.4	69.74	100.0	3.69	6.22	41.35	100.0
head	78.0			6.22			

BGE AS-RECEIVED ASH - MICROTRAC ANALYSIS		
Channel	Cumulative	Volume
62	100.0	3.8
44	96.2	12.1
31	84.1	15.7
22	68.4	16.4
16	52.0	14.9
11	37.1	11.5
7.8	25.6	8.8
5.5	16.7	6.1
3.9	10.6	4.4
2.8	6.2	3.9
1.9	2.3	1.5
1.4	0.8	0.8
0.9	0.0	0.0

AEP AS-RECEIVED ASH - SIZE DISTRIBUTION and LOI by Size							
Tyler Mesh		% Weight		% LOI		Distribution	
	Wt. (g)	individual	cumulative	individual	cumulative	individual	cumulative
+65	0.6	0.77	0.77	26.81	26.81	0.95	0.95
+100	1.4	1.79	2.55	56.99	47.94	4.70	5.64
+150	4.2	5.36	7.91	48.84	48.55	12.08	17.72
+200	7.8	9.95	17.86	43.11	45.52	19.80	37.52
+270	8.0	10.20	28.06	31.52	40.43	14.85	52.36
+325	3.8	4.85	32.91	32.66	39.28	7.31	59.67
+400	10.4	13.27	46.17	22.53	34.47	13.79	73.47
-400	42.2	53.83	100.0	10.68	21.66	26.53	100.0
head	78.4			21.66			

AEP AS-RECEIVED ASH - MICROTRAC ANALYSIS		
Channel	Cumulative	Volume
62	100.0	5.2
44	94.8	15.2
31	79.6	18.1
22	61.5	16.2
16	45.3	14.3
11	31.0	10.9
7.8	20.1	8.2
5.5	11.9	5.1
3.9	6.8	3.2
2.8	3.6	2.7
1.9	0.9	0.7
1.4	0.2	0.2
0.9	0.0	0.0

APPENDIX B

TASK 2.3 Laboratory Testing of Ash Processing Operations

Summary of Laboratory Test Results

SUMMARY OF LABORATORY FLOTATION RESULTS

Test #	Ash Type	Collector		Frother		Clean Ash		Carbon Concentrate			Screen Fraction		Middlings		Remarks
		Type	#/T	Type	#/T	%LOI	%wt rec.	%wt	%LOI	%LOI rec.	%wt	%LOI	%wt	%LOI	
1	NPC	mix	2.0	DF	1.54										
2	NPC	mix	2.0	DF	1.45										
3	AEP	mix	3.0	DF	2.8	0.86	55.8	32.9	61.8	87.5	-	-	11.3	21.5	One cleaner stage
4	BGE	mix	2.0	DF	1.35	5.96	75.6	14.5	13.9	28.1	-	-	9.86	6.72	One cleaner stage
5	AEP	mix	3.0	DF	2.8	12.85	74.0	3.9	74.0	12.5	-	-	22.15	47.4	Five cleaner stages
6	AEP	mix	3.75	DF	3.13	1.37	56.4	30.0	51.7	67.6	-	-	13.6	49.0	One scavenger stage, add frother just prior to flot
7	AEP	mix	3.0	DF	2.99	7.27	63.5	22.9	61.8	58.4	-	-	13.6	40.2	Repeat T3 w/ new collector mix
8	AEP	mix	3.0	DF	2.89	15.63	75.7	24.3	43.1	46.9	-	-	-	-	Repeat T7, shorter conditioning time
9	AEP	mix	3.50	DF	2.89	6.24	62.6	37.4	40.5	79.5	-	-	-	-	Repeat T7, more collector, no cleaners
10	AEP	mix	2.5	DF	2.89	12.18	70.0	30.0	48.3	62.9	-	-	-	-	Repeat T9, less collector, no cleaners
11	FA5	mix	1.75	DF	1.35	0.40	70.5	29.5	18.0	94.9	-	-	-	-	baseline for % solids test, 8.09% solids
12	FA5	mix	1.75	DF	1.35	0.35	69.1	30.9	17.2	95.6	-	-	-	-	Repeat T11, 15.28% solids
13	FA5	mix	1.75	DF	1.35	0.51	73.0	27.0	18.8	93.8	-	-	-	-	Repeat T12, 20% solids
14	FA5	mix	1.75	DF	1.35	0.55	65.4	19.5	23.0	79.0	-	-	15.0	5.5	Repeat T13, 25% solids, collected 2 froths
15	FA5	mix	1.75	DF	1.35	0.62	66.0	17.6	21.2	66.3	-	-	16.4	9.1	Repeat T14, 30% solids
16	FA5	mix	1.75	DF	1.35	0.80	64.0	18.8	17.7	58.1	-	-	17.2	11.0	Repeat T15, 35% solids
17	FA5	mix	1.75	DF	1.35	1.53	71.2	16.1	17.2	49.9	-	-	12.7	13.2	Repeat T16, 40% solids
18	AEP	mix	3.0	DF	3.76	6.69	61.5	38.5	50.6	82.6	-	-	-	-	separate conditioning, adjust pH w/ Na2CO3
19	AEP	mix	3.0	DF	3.76	9.95	63.2	36.8	48.4	74.0	-	-	-	-	Repeat T18, pH to 9
20	AEP	mix	3.0	DF	3.76	8.4	67.0	33.0	53.8	76.0	-	-	-	-	No Na2SiO4, used Na2CO3 H2SO4 for pH
21	AEP	mix	3.5	DF	3.76	6.91	70.5	29.5	62.6	79.1	-	-	-	-	Repeat T20 w/ increased collector

SUMMARY OF LABORATORY FLOTATION RESULTS

Test #	Ash Type	Collector		Frother		Clean Ash		Carbon Concentrate			Screen Fraction		Middlings		Remarks
		Type	#/T	Type	#/T	%LOI	%wt rec.	%wt	%LOI	rec.	%wt	%LOI	%wt	%LOI	
22	BGE	mix	2.0	DF	1.35	14.53	70.3	29.7	3.6	9.4	-	-	-	-	
23	AEP	MAS	8.02	DF/P	1.25/2	4.88	67.2	30.6	62.6	82.0	2.2	42.7	-	-	flot - 100m fraction, modify reagent scheme
24	AEP	MAS	9.0	DF/P	1.67/2	1.46	61.4	30.8	69.0	90.7	2.2	49.6	5.7	3.8	repeat T23, w/ cleaner flot, increased collector
25	AEP	MAS	10.0	DF/P	2.3/2	2.22	63.4	29.7	68.0	88.2	2.5	45.4	4.4	3.4	Repeat T24, increased collector
26	AEP	MAS	10.0	DF/P	1.67/2	0.35	57.1	28.5	74.1	93.3	2.4	44.5	11.9	5.0	Repeat T25 w/ dif. flot cell, stage add frother
27	AEP	mix	10.0	DF/P	1.25/2	0.40	54.5	42.3	51.1	92.9	3.2	44.8	-	-	Repeat T26 w/ different collector
28	AEP	MAS	10.0	DF	1.25	0.18	45.6	51.2	42.6	94.0	3.1	42.4	-	-	DF frother only